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22nd Symposium on Application of Plasma Processes
and
11th EU-Japan Joint Symposium on Plasma Processing

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18-24 January, 2019

Edited by V. Medvecká, J. Országh, P. Papp, Š. Matejčík

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NEW AND VERSATILE MINATURE MICROWAVE PLASMA SOURCE

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Miniature Microwave Inductively Coupled Plasma (MMWICP) source is characterized by means of Optical Emission Spectroscopy (OES) in nitrogen gas flow, which gives the information on basic plasma properties. Depending on the incident power the discharge runs in E-mode or in more efficient H-mode. The high resolution radial images of the source reveal different morphologies of different discharge modes. The measurements show an unexpected limitation in dissipated power, accompanied by spontaneous transition from H- to E-mode. The efficiency of the source is high: about 67% of incident power (P_0) is deposited in the discharge, which is estimated from OES.

1. Experimental set-up

MMWICP source consist of a quartz tube with the outer diameter of 7 mm and 1 mm thick walls (see figure 1). Through the quartz tube a continuous flow of 150 sccm (standard cubic centimetre per minute) of N_2 is running. While the source operates in the wide range from few tenths of Pa to atmospheric pressure, during the reported experiment the gas pressure is kept at 1000 Pa. The microwave power is coupled through a copper resonator, which combine an inductive loop and a gap capacitor unified in a single copper block. The resonator is tuned to the resonance frequency of 2.6 GHz. The MW power is provided by a signal generator with the maximum delivered power of 250 W. The generator is equipped with an integrated directional coupler and two power meters, measuring the forwarded and reflected powers. The further details are provided in [1].

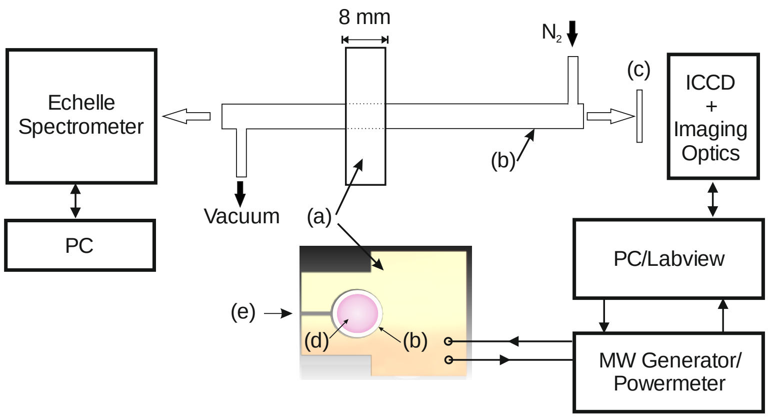

Fig. 1. Schematic of the experimental set-up for characterization of MMW plasma source. Top: optical arrangement; bottom: copper resonator cross section perpendicular to the optical axis. (a) copper resonator, (b) quartz tube, (c) interference filter (d) plasma and (e) gap capacitor.

The OES is conducted by collecting light emission perpendicular to the discharge cross-section. A calibrated Echelle spectrometer measures absolute intensity of two nitrogen

emission lines: N_2(C,0-0) and N_2^+(B,0-0). The population/depopulation of N_2 (C) and N_2^+(B) states is well described and previously used to determine the plasma conditions in different sources [2, 3]. In the combination with a simple collision- radiative model, the outcome of OES is line integrated plasma density n_e, electron velocity distribution function (EVDF) and normalized electrical field strength *E/N*. The analysis of the vibrational spectra gives rotational temperatures of N_2 and N_2^+, respectively. Since the electronic impact excitation of diatomic molecules is limited by the selection rule $\Delta J = 0, \pm 1$, the rotational distribution in the excited molecular state is approximately equal to the rotational distribution in the ground state of the molecule. This allows to determine a gas temperature T_g in the plasma by using the rotational distribution in the emission spectrum of the N_2(C-B,0-0) vibrational band.
Simultaneously to OES, a calibrated Intensified Charge-Coupled Device (ICCD) camera is recording the radial distribution of plasma emission. Two narrow band-pass interference filters can be used to separate two different emission lines: N_2(C,0-0) and N_2^+(B,0-0). By using the same analysis as by space averaged OES, the radially resolved line integrated n_e and *E/N* are determined.

2. Results

The MMWICP characteristics depend strongly on the absorbed power P_a. For the sake of comparison two different absorbed powers are chosen, corresponding to the different discharge modes: hybrid mode (E/H) at P_a = 12 W and H-mode at P_a = 80 W. The OES reveals different plasma densities and gas temperatures of both modes. The H-mode has n_e = 3.5×10^{19} m^{-3} and T_g = 1600 K, which is expected by high efficient inductive coupling. At low powers the discharge still reaches relatively high plasma density of $n_e = 6.8\times10^{18}$ m^{-3}.

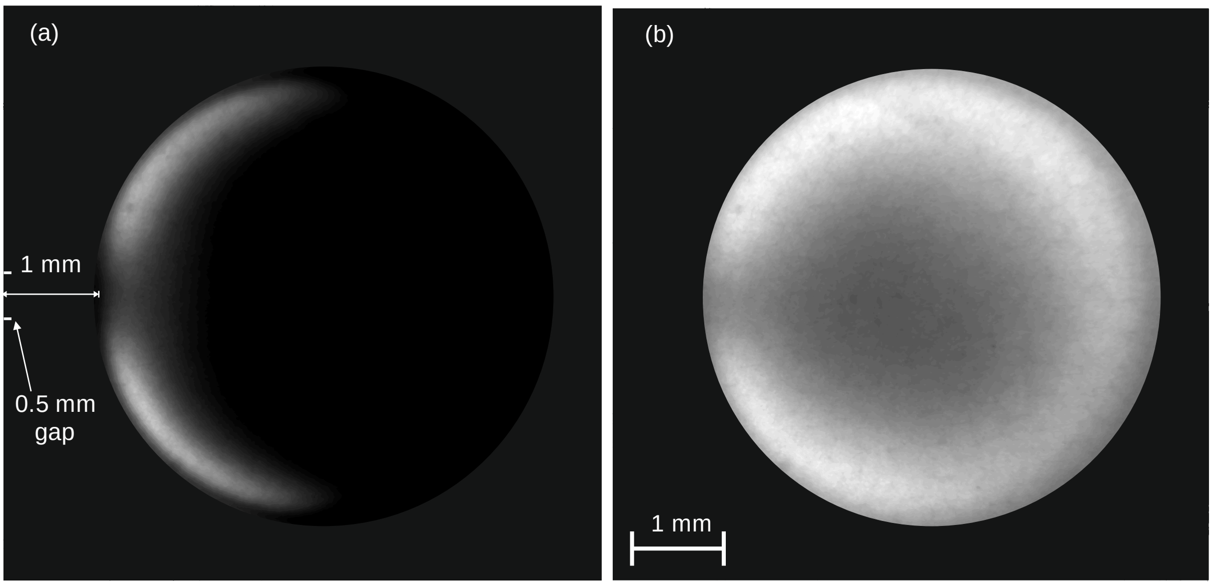

Fig. 2. Radial images of the MW plasma: (a) hybrid E/H mode, absorbed power P_{abs} = 12 W and (b) H - mode, P_{abs} = 78 W. Two horizontal bars on (a) indicate the approximate position of the 0.5 mm gap capacitor, situated closely to the outer quartz tube wall, e.g. 1 mm away from the plasma to the left. Light reflection by the tube wall is suppressed. (Reproduced from [1]).

Fig. 2 presents two high-resolution images of a discharge cross-section at two different absorbed powers: (a) 12 W and (b) 78 W. These conditions corroborate with two different discharge modes. At low powers the discharge is concentrated in the vicinity of the gap capacitor, indicating strong capacitive coupling, which is characteristic for the E-mode. Nevertheless, the high plasma density and the discharge morphology suggests the existence of

hybrid E/H mode rather than pure E-mode. The gas temperature in this hybrid more is about 650 K. The H-mode morphology reassembles a “donut” shape. The discharge emission peaks at the vicinity of the tube wall and have a ring form according to the circular pattern of the current flow by inductive power coupling.
The narrow band interference filters in front of the camera objective isolate the spatially resolved absolute light intensities either of N_2(C,0-0) or N_2^+(B,0-0) over the discharge cross-section. By using the same analysis as by OES the spatially resolved n_e and E/N are recorded. The dissipated power P_{diss} density in the plasma can be estimated by

$$\frac{P_{diss}}{V} = n_e e v_d \left(\frac{E}{N} \right) N \ . \qquad (1)$$

V is the plasma volume, e – the elementary charge, v_d – the electron drift velocity and N the gas density. The integration over the plasma volume gives a value of dissipated power in the H – mode about P_{diss} = 52 W. The comparison with P_a by electrical measurements, which include the power absorbed in connectors, cables, copper resonator and plasma, gives relative good agreement.
One of the intriguing characteristics of the MMWICP source is the *limitation in the absorbed power*. We have to note that, by increasing the incident power for given pressure and reactor radius there is a limitation at 158 W. By further increase, the plasma cannot support the H-mode and switch to the E-mode or the hybrid mode. The reson for this behavior is still not clearly understood and needs further experimental and theoretical studies. Nevertheless, some preliminary results on the spontaneous transition form H- to E-mode for the pressure of p = 470 Pa in nitrogen are shown on Fig. 3.

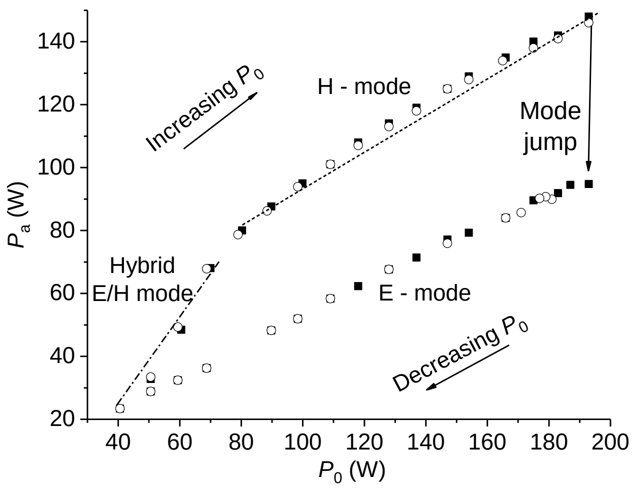

Fig. 3. Absorbed MW power P_a vs. incident power P_0. Different points indicate two cycles of power variation. Different discharge modi are indicated: H-mode, E-mode and hybrid (E/H) mode. For the given pressure of 470 Pa the maximum absorbed power is P_a = 148 W. The MW frequency is f = 2.465 GHz and is kept constant during the measurements. The stright lines are arbitrary and are used to indicate different slopes of power change in hybrid- and H- mode.

The discharge is ignited in hybrid (E/H) mode at incident power P_0 = 40 W. By increasing the incident power, the absorbed power follows and from P_0 > 80 W the discharge is running in H- mode. By reaching some critical value (about P_a = 148 W for 470 Pa and P_a = 158 W for 1000 Pa) the discharge switch to E- mode spontaneously (indicated as a “Mode jump” at Fig. 3). By decreasing P_0 the discharge runs through the lower brunch of the hysteresis. Only at low incident powers it is than possible to run the discharge through upper hysteresis’s brunch again. Two consecutive cycles of measurements (solid and opened symbols in figure 3) show high degree of reproducibility.

The novel MMWICP source operated in nitrogen flow at 1000 Pa is characterized by optical emission spectroscopy and optical imaging. The absolute intensities of the second positive and the first negative emission systems of molecular nitrogen are measured. The careful analysis of the rotational distributions of N_2(C-B,0-0) and of N_2^+(B-X,0-0) vibrational bands helps to rule out the stepwise excitation as the dominate excitation mechanism for the N_2^+(B-X) state. The absolute intensity measurements detect the high electron density (3.5×10^{19} m^{-3}) and high gas temperature (1600 K). Optical imaging reveals different emission patterns for H – mode and hybrid – mode. Additionally, the spatially resolved optical emission is used to obtain the distributions of electron density and electric field over the discharge cross-section. The relatve high discharge efficiency is measured: about 67% of the incident power is consumed in the H-mode. Some limitations in the maximum of the absorbed power is detected and deserve further analysis. The preliminary mesurements on atmospheric pressure discharge in argon indicates that the MMWICP source can be widely used for industrial and other applications such as plasma medicine and biotechnologies.

3. References

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